
फॉस्फोरिक एसिड, खाद्य ग्रेड — विशिष्टि
(दूसरा पुनरीक्षण)

Phosphoric Acid, Food Grade —
Specification
(Second Revision)

ICS 67.220.20

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FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Food Additives Sectional Committee had been approved by the Food and Agriculture Division Council.

With the increased production of processed foods, manufacturers have started adding a large number of substances, generally in small quantities, to improve the appearance, flavor, and texture or storage properties of the processed foods. As impurities in these substances have been found to be harmful, it is necessary to have a strict quality control of these food additives. Phosphoric acid is used as an acidulant and is permitted under the *Food Safety and Standards Act, 2006* and Rules framed thereunder. This standard would help in checking purity which requires to be checked at the stage of manufacture, for it is extremely difficult to detect the impurity once these substances are added to the processed foods.

This standard was first published in 1983 based on the then existing JECFA 'Specification and food chemical codex' of USA. The first revision of this standard was taken up in 2007 wherein the limit for nitrates, volatile acids, chlorides and sulphates were included to align it with the international requirements along with the revision and updation of methods of test.

In the second revision of this standard, the latest publication for phosphoric acid issued by JECFA has been taken into consideration and requirements have been aligned with *Food Safety and Standards Act, 2006*. Heavy metals, such as copper, tin and cadmium have been added along with their instrumental methods of test. Further, molybdenum and aluminum have also been included to avoid spent phosphoric acid which is being used in food industry.

In the preparation of this standard, due consideration has been given to the *Legal Metrology (Packaged Commodities) Rules, 2011* and *Food Safety and Standards Act, 2006* and Rules framed thereunder. The standard is however, subject to restrictions imposed under these rules wherever applicable.

The composition of the committee responsible for the formulation of this standard is stipulated in Annex H.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

PHOSPHORIC ACID, FOOD GRADE — SPECIFICATION

(Second Revision)

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for phosphoric acid, food grade.

2 REFERENCES

The following standards contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>IS No.</i>	<i>Title</i>
798 : 2020	Orthophosphoric Acid — Specification (<i>third revision</i>)
1070 : 1992	Reagent grade water (<i>third revision</i>)
1699 : 1995	Methods of sampling and test for food colours (<i>second revision</i>)

IS No.

Title

2491 : 2013 Food hygiene — General principles — Code of practice (*second revision*)

3 REQUIREMENTS

3.1 Description

Phosphoric acid shall be a clear, colourless, odourless viscous liquid. It shall be miscible in water and ethanol. Phosphoric acid is strong acidic, even after high dilution.

3.2 Test for Phosphate

Neutralize a few millilitres of phosphoric acid and add dilute nitric acid then add an equal volume of ammonium molybdate solution and warm. A bright canary-yellow precipitate is obtained which is soluble in dilute ammonia.

3.3 The material shall also conform to the requirements given in Table 1.

Table 1 Requirements for Phosphoric Acid

(Clause 3.3)

Sl No.	Characteristics	Requirement	Method of Test, Ref to	
			Annex of this Standard	Clause
(1)	(2)	(3)	(4)	(5)
i)	Purity as H_3PO_4 , percent by mass, <i>Min</i>	85	A	-
ii)	Nitrates, mg/kg, <i>Max</i>	5	B	-
iii)	Volatile acids, mg/kg, <i>Max</i>	10	C	-
iv)	Chlorides, mg/kg, <i>Max</i>	200	D	-
v)	Sulphates percent by mass, <i>Max</i>	0.15	E	-
vi)	Fluoride, mg/kg, <i>Max</i>	10	F	-
vii)	Arsenic (as As), mg/kg, <i>Max</i>	2	-	15 of IS 1699
viii)	Lead (as Pb), mg/kg, <i>Max</i>	4	-	A-10 and A-20 IS 798
ix)	Copper (as Cu) mg/kg, <i>Max</i>	30	G	-
x)	Tin, mg/kg, <i>Max</i>	250	G	-
xi)	Cadmium(as Cd), mg/kg, <i>Max</i>	1.50	G	-
xii)	Mercury, mg/kg, <i>Max</i>	1.00	-	A-17 and A-20 of IS 798
xiii)	Molybdenum (as Mo), mg/kg, <i>Max</i>	10	-	A-16 and A-20 of IS 798
xiv)	Aluminium (as Al), mg/kg, <i>Max</i>	10	-	A-18 and A-20 of IS 798

3.4 The material shall be processed, packed, stored and distributed under hygienic conditions in licensed premises (*see* IS 2491).

4 PACKING, STORAGE AND MARKING

4.1 Packing

The material shall be securely packed in well-filled containers with minimum access to air. The containers shall be, such as to preclude contamination of the contents with metals or other impurities.

4.2 Storage

The material shall be stored in a cool and dry place so as to avoid excessive exposure to heat.

4.3 Marking

4.3.1 Each container shall be legibly and indelibly marked with the following information:

- a) Name of the material including the words 'Food Grade';
- b) Name and address of the manufacturer;
- c) Date of manufacture;
- d) Batch or Code number;
- e) Net content when packed;
- f) Phosphoric acid content;
- g) Instruction for storage;
- h) Best before date (Month and Year to be given by the manufacturer); and
- j) Any other requirements as given under the Standards of Weights and Measures (Packaged Commodities) Rules, 1977 and Food Safety and Standards (Contaminants, Toxins and Residues) Regulations 2011

4.3.2 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations framed thereunder and the products may be marked with the Standard Mark.

5 SAMPLING

Representative samples of the material shall be drawn according to the method prescribed in 4 of IS 1699.

6 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the experimental results.

ANNEX A

[Table 1, Sl No. (i)]

DETERMINATION OF PURITY**A-1 REAGENTS**

A-1.1 Thymolphthalein Solution — Dissolve 0.100 g of in 100 ml of thymolphthalein ethanol and filter, if necessary.

A- 1.2 Sodium Hydroxide — 1.0 N.

A-2 PROCEDURE

Weigh 1.000 g of phosphoric acid into a glass-stoppered flask, dilute with about 100 ml of water, add 0.5 ml of thymolphthalein solution. Titrate with 1N sodium hydroxide. Each millilitre of 1N sodium hydroxide is equivalent to 0.049 g of H_3PO_4 .

ANNEX B

[Table 1, Sl No. (ii)]

DETERMINATION OF NITRATE**B-1 PROCEDURE**

Dilute 3.48 g of the sample in 10 ml with water and

add mg of sodium chloride, 0.1 ml of indigo carmine solution, and add 10 ml of sulphuric acid. The blue colour shall not disappear entirely within 5 min.

ANNEX C

[Table 1, Sl No. (iii)]

DETERMINATION OF VOLITILE ACIDS**C-1 PROCEDURE**

Dilute 60.05 g of the sample with 75 ml of freshly boiled and cooled water in a distilling flask with a spray tap, and distill 50 ml. To the distillate add phenolphthalein

and titrate with 0.1 N sodium hydroxide. Not more than 0.1 ml of 0.1 N sodium hydroxide shall be required for neutralization.

ANNEX D

[Table 1, Sl No. (iv)]

TEST FOR CHLORIDES**D-1 PROCEDURE**

Place 1.78 g of the sample in a Nessler tube, dissolve it in about 30 ml of water, and neutralize with dilute nitric acid TS. If the solution is alkaline. Add 6 ml of dilute nitric acid TS and dilute to 50 ml with water. If the use of a sample solution is prescribed, transfer the sample solution into a Nessler tube and dilute to 50 ml with water. Transfer 1 ml of 0.01 N hydrochloric acid into another Nessler tube to serve as the standard, add 6 ml

of dilute nitric acid TS, and dilute to 50 ml with water.

If the solution containing the sample is not clear, filter both solutions under the same conditions. Add 1 ml of silver nitrate TS to each solution, mix thoroughly, and allow to stand for 5 min protected from direct sunlight. Compare the turbidity of the two solutions by observing the Nessler tubes from the sides and the tops against a black background. The turbidity of the sample solution shall not exceed that of the standard.

ANNEX E

[Table 1, SI No. (v)]

TEST FOR SULPHATES

E-1 PROCEDURE

Place 1.25 g of the sample in a Nessler tube, dissolve it in about 30 ml of water, and neutralize with dilute hydrochloric acid TS, if the solution is alkaline. Add 1 ml of dilute hydrochloric acid TS and dilute to 50 ml with water. If the use of a sample solution is prescribed, transfer the sample solution into a Nessler tube and dilute to 50 ml with water. Transfer the prescribed volume of 0.01 N sulphuric acid into another Nessler

tube to serve as the standard, add 1 ml of dilute hydrochloric acid TS, and dilute to 50 ml with water.

If the solution containing the sample is not clear, filter both solutions under the same conditions. Add 2 ml of barium chloride TS to each solution, mix thoroughly, and allow to stand for 10 min. Compare the turbidity of the two solutions by observing the Nessler tubes from the sides and the tops against a black background. The turbidity of the sample does not exceed that of the standard.

ANNEX F

[Table 1, SI No. (vi)]

DETERMINATION OF FLUORIDE

F-1 APPARATUS

Assembly as shown in Fig. 1.

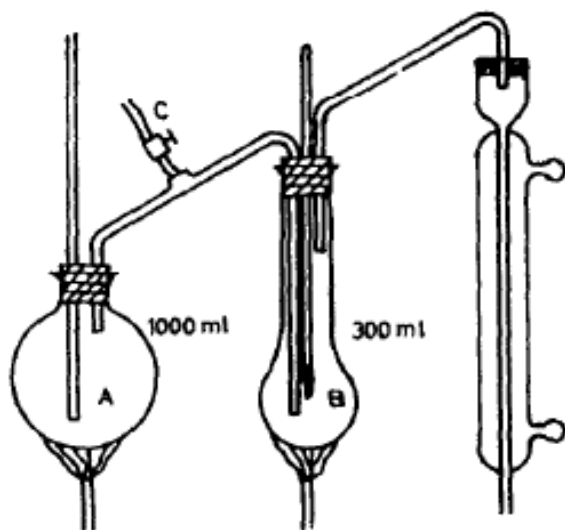


FIG. 1 APPARATUS FOR LIMIT TEST FOR FLUORIDE

F-2 REAGENTS

F-2.1 Phenolphthalein Solution — Dissolve 0.2 g of phenolphthalein in 60 ml of 90 percent ethanol and sufficient water to make 100 ml.

F-2.2 Sodium Hydroxide — 1 N.

F-2.3 Sulphuric Acid — Concentrated.

F-2.4 Sodium Fluoride Solution — Containing 50 µg of fluoride.

F-2.5 Hydrochloric Acid — 4 N.

F-2.6 Zirconium Alizarin Solution

Dissolve 0.80 g of zirconium nitrate in water, add a few drops of 4 N nitric acid and make up to 100 ml with water. Dissolve 0.10 g of alizarin sulphonate monohydrate in 20 ml of water, make up to 100 ml with ethanol. Mix 1 ml of first solution with 1 ml of second solution and add 18 ml of water. The solution should be clear and the dilution should be freshly prepared.

F-3 PROCEDURE

F-3.1 Place about 500 ml of water in flask (see Fig. 1), make it alkaline to phenolphthalein solution with 1 N sodium hydroxide and heat the water to boiling. Leave open the screw clamp at C.

F-3.2 Determine the appropriate weight of the sample (W , in gram) by the formula $W = 50/L$, in which L is the fluoride limit, in mg/kg. Accurately weigh the calculated amount of the sample, place it in flask B, and add 10 ml of water to the flask. Add 17 ml of sulphuric acid slowly down the sides of the flask so that it forms a layer under the water. Connect flask B to the apparatus. Place the tip of the condenser into a flask containing 5 ml of water. Mix the contents of flask B, heat to 150 °C and slowly shut the screw clamp at C. Regulate the temperature of the solution in the flask B to 150-153 °C during the distillation. Continue until 70 ml of distillate have been collected.

F-3.3 Place the distillate in a 100 ml Nessler tube. Place 80 ml of sodium fluoride solution, containing 50 µg of fluorine, in a second Nessler tube. To each tube add 8.5 ml of 4N hydrochloric acid and 2.0 ml of zirconium

alizarin solution and makeup to 100 ml with water. Let the tubes stand for 15 min.

F-3.4 The colour of the test solution containing the sample shall not be darker than that of the standard solution.

ANNEX G

DETERMINATION OF CUPPER, TIN AND CADMIUM BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD

G-1 PRINCIPLE

The solution under analysis is passed with the help of a peristaltic pump through a nebulizer inside a spray chamber. The aerosol formed is aspirated to argon plasma where the atoms are excited. These excited atoms then return back to the lower energy state by emitting radiation of specific wavelength. These emitted radiations are characteristic of an element and are measured by the Photomultiplier tube detector and wavelength/intensity of emitted radiation is directly proportional to the concentration of respective constituent element in the sample.

G-2 REAGENTS AND SOLUTIONS

G-2.1.1 Nitric Acid (65 Percent) Suprapure

G-2.1.2 Standard Stock Solution — Prepared or use commercially available certified stock solution of 10, 100 or 1000 µg/ml of lead (and/or iron, calcium, magnesium, manganese, arsenic, molybdenum, aluminium and mercury) in 2-5 percent nitric acid.

G-2.1.3 Standard Solution — Pipette out 5 ml from 100 µg/ml standard stock solution into a 100 ml volumetric flask and make up with 2-5 percent nitric acid to prepare 5 µg/ml solution. From this 5 µg/ml solution take 1.0, 3.0 and 5.0 ml in 50 ml volumetric flask and make up volume with 2-5 percent nitric acid to prepare 0.1, 0.3 and 0.5 µg/ml of copper, (and/or tin and cadmium).

G-2.1.4 Sample Preparation — Weigh about 2.5 g phosphoric acid sample in a 50 ml volumetric flask and add 1.0 ml Nitric acid and make up the volume with water and analyse the sample using ICP-OES.

G-3 INSTRUMENT

Set up the instrument as per the manufacturer's instructions. Set ICP-OES method to 327.396 nm for copper (235.485 nm for tin, 214.441 nm for cadmium) with axial mode.

For mercury analysis instrument set to hydride mode and other instrumental condition same as above.

G-4 PROCEDURE

Set up the instrumental method to measure the intensities of the 0.1, 0.3 and 0.5 µg/ml standard solutions. The calibration curve for lead (and/or iron, calcium, magnesium, manganese and arsenic) should be linear. Examine the spectra of the element and make any necessary adjustments to the exact peak positions and baselines to ensure proper measurements of the respective peak intensities. Analyze the sample solution and calculate the concentration in µg/ml of the copper (and/or tin and cadmium) in the sample solution.

G-5 CALCULATION

Calculate the quantity in mg/kg of the copper (and/or tin and cadmium) in the sample by multiplying this value by 20 (dilution factor).

ANNEX H

(Foreword)

COMMITTEE COMPOSITION

Food Additives Sectional Committee, FAD 08

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SCIENTIST 'C' (FAD), BIS

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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